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- (71) Applicant: **KIMBERLY-CLARK WORLDWIDE, INC.** [US/US]; 401 N. Lake Street, Neenah, WI 54956 (US).
- (72) Inventors: **ABUTO, Francis, Paul**; 515 Oakmont Hill, Duluth, GA 30097 (US). **FAIRBANKS, Jason, Sybren**; 7875 Trailside Way, Gainesville, GA 30506 (US). **SCHLEINZ, Alan, Francis**; 1312 West Cedar Street, Appleton, WI 54914 (US). **SMITH, JR., Roland, Columbus**; 2771 Pierce Road, Gainesville, GA 30507 (US). **SOERENS, Dave, Allen**; 736 Kensington Road, Neenah, WI 54956 (US). **TOPOLKARAEV, Vasily, Aramovich**; 1730 S. Lee Street, Appleton, WI 54915 (US). **WALLAJAPET, Palani, Raj Ramaswami**; 808 Kensington Road, Neenah, WI 54956 (US). **WORKMAN, JR., Jerome, James**; 1900 South Lee Street, Appleton, WI 54915 (US). **ZHOU, Peiguang**; N362 Tamarack Drive, Appleton, WI 54915 (US).
- (49) Agents: **FLACK, Steven, D. et al.**; KIMBERLY-CLARK WORLDWIDE, INC., 401 N. Lake St., Neenah, WI 54956 (US).
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(54) Title: TARGETED BONDING FIBERS FOR STABILIZED ABSORBENT STRUCTURES

(57) Abstract: A fiber is provided wherein the fiber contains an energy receptive additive. The fiber provides rapid heating when subjected to dielectric energy such as radio frequency or microwave radiation. The energy receptive additive has a dielectric loss of between about 0.5 and 15, preferably between 1 and 15 and most preferably between 5 and 15. The fibers may be crimped, extensible or elastic.

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TARGETED BONDING FIBERS FOR STABILIZED ABSORBENT STRUCTURES

BACKGROUND OF THE INVENTION

5 This invention relates generally to nonwoven fibers made from a thermoplastic resin.

Thermoplastic resins have been extruded to form fibers, fabrics and webs for a number of years. Common thermoplastics for this application are polyolefins, particularly polyethylene and polypropylene. Other materials such as polyesters, polyetheresters, polyamides and polyurethanes are also used to form nonwoven fabrics.

10 Nonwoven fabrics or webs are useful for a wide variety of applications such as personal care products, towels, recreational or protective fabrics and as geotextiles and filter media. The nonwoven fibers used in these applications may be made by many processes known in art, such as spunbonding and meltblowing. Nonwoven fibers may be processed into webs through bonding and carding processes, airforming (airlaying), and other
15 processes.

Fibers are commonly consolidated to form a web by adhesive bonding, mechanical bonding such as hydroentangling and by ultrasonic and thermal bonding processes using bonding fibers. These processes work well but have individual idiosyncratic drawbacks. Adhesive bonding and hydroentangling, for example, often necessitate the removal of water,
20 a process involving the addition of more energy for a rather lengthy time. Ultrasonic bonding is also an energy intensive activity involving energy being added somewhat randomly to a web. Thermal bonding also involves the addition of energy to the web in a relatively slow process to melt particular binder fibers.

A process wherein the binder fibers could be heated more rapidly to their melting
25 temperature would be very advantageous for high-speed industrial applications. An improved binder fiber that could reach its melting temperature under the proper conditions in

a matter of fractions of a second would significantly reduce processing and production time and thus increase manufacturing efficiency and reduce product cost. Such binder fibers would probably also use less energy than fibers that more slowly reached their melting temperature since less energy would probably be absorbed by adjacent materials. It is an
5 object of this invention to produce such a fiber.

SUMMARY OF THE INVENTION

The objects of this invention are achieved by a fiber having an energy receptive
10 additive capable of dielectric heating and having a dielectric loss (as defined below) of between 0.5 to 15. The fiber more particularly should have a dielectric loss of at least 1 and still more particularly have a dielectric loss of at least 5. It is preferred that the fiber have a dielectric loss tangent (as defined below) of between 0.1 to 1, more particularly between 0.3 to 0.7.

15 The fiber may be made from a synthetic polymer selected from groups such as polyolefins, polycaprolactones, polyamides, polyetheramides, polyurethanes, polyesters, Poly (meth) acrylates metal salts, polyether, poly(ethylene- vinyl acetate) random and block copolymers, polyethylene -b- polyethylene glycol block copolymers, polypropylene oxide-b-polyethylene oxide copolymers and blends thereof.

20 The energy receptive additive may be, for example, carbon black, magnetite, silicon carbide, calcium chloride, zircon, ferrite, tin oxide, silicon carbide, calcium chloride, alumina, magnesium oxide, and titanium dioxide. The energy receptive additive may be present in an amount between 2 and 40 weight percent, more particularly between 5 and 15 weight percent, of the total fiber weight.

The fiber may also be a bicomponent fiber of the sheath/core or island in the sea type. The energy receptive additive may be present in the sheath or core of a sheath/core type bicomponent fiber.

The fiber may also be a biconstituent fiber. The fiber may be crimped, extendible
5 and/or elastic and the energy receptive additive may be carbon black in an amount between 2 and 40 weight percent.

A particular embodiment is a fiber having an energy receptive additive in an amount between 5 and 15 weight percent, synthetic polymer and having a dielectric loss of at least 0.5.

10 Nonwoven webs may be produced with the fibers of this invention as well.

DEFINITIONS

As used herein the term "nonwoven fabric or web" means a web having a structure
15 of individual fibers or threads which are interlaid, but not in an identifiable manner as in a knitted fabric. Nonwoven fabrics or webs have been formed from many processes such as for example, meltblowing processes, spunbonding processes, and bonded carded web processes. The basis weight of nonwoven fabrics is usually expressed in ounces of material per square yard (osy) or grams per square meter (gsm) and the fiber diameters useful are
20 usually expressed in microns. (Note that to convert from osy to gsm, multiply osy by 33.91).

As used herein the term "meltblown fibers" means fibers formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into converging high velocity, usually hot, gas (e.g. air) streams which attenuate the filaments of molten thermoplastic material to reduce their diameter,
25 which may be to microfiber diameter. Thereafter, the meltblown fibers are carried by the high velocity gas stream and are deposited on a collecting surface to form a web of

randomly dispersed meltblown fibers. Such a process is disclosed, for example, in US Patent 3,849,241 to Butin et al. Meltblown fibers are microfibers which may be continuous or discontinuous, are generally smaller than 10 microns in average diameter, and are generally tacky when deposited onto a collecting surface.

5 "Spunbonded fibers" refers to small diameter fibers that are formed by extruding molten thermoplastic material as filaments from a plurality of fine capillaries of a spinneret. Such a process is disclosed in, for example, US Patent 4,340,563 to Appel et al. and US Patent 3,802,817 to Matsuki et al. The fibers may also have shapes such as those described, for example, in US Patents 5,277,976 to Hogle et al. which describes fibers with
10 unconventional shapes.

 As used herein the term "bicomponent fibers" refers to fibers which have been formed from at least two polymers extruded from separate extruders but spun together to form one fiber. Bicomponent fibers are also sometimes referred to as multicomponent or conjugate fibers. The polymers are usually different from each other though bicomponent
15 fibers may be monocomponent fibers. The polymers are arranged in substantially constantly positioned distinct zones across the cross-section of the bicomponent fibers and extend continuously along the length of the bicomponent fibers. The configuration of such a bicomponent fiber may be, for example, a sheath/core arrangement wherein one polymer is surrounded by another or may be a side by side arrangement, a pie arrangement or an
20 "islands-in-the-sea" arrangement. Bicomponent fibers are taught in US Patent 5,108,820 to Kaneko et al., US Patent 4,795,668 to Krueger et al., US Patent 5,540,992 to Marcher et al. and US Patent 5,336,552 to Strack et al. Bicomponent fibers are also taught in US Patent 5,382,400 to Pike et al. and may be used to produce crimp in the fibers by using the differential rates of expansion and contraction of the two (or more) polymers. For two
25 component fibers, the polymers may be present in ratios of 75/25, 50/50, 25/75 or any other desired ratios. The fibers may also have shapes such as those described in US Patents

5,277,976 to Hogle et al., US Patent 5,466,410 to Hills and 5,069,970 and 5,057,368 to Largman et al., which describe fibers with unconventional shapes.

As used herein the term "biconstituent fibers" refers to fibers which have been formed from at least two polymers extruded from the same extruder as a blend. The term "blend" is defined below. Biconstituent fibers do not have the various polymer components arranged in relatively constantly positioned distinct zones across the cross-sectional area of the fiber and the various polymers are usually not continuous along the entire length of the fiber, instead usually forming fibrils or protofibrils which start and end at random. Biconstituent fibers are sometimes also referred to as multiconstituent fibers. Fibers of this general type are discussed in, for example, US Patents 5,108,827 and 5,294,482 to Gessner.

Bicomponent and biconstituent fibers are also discussed in the textbook Polymer Blends and Composites by John A. Manson and Leslie H. Sperling, copyright 1976 by Plenum Press, a division of Plenum Publishing Corporation of New York, ISBN 0-306-30831-2, at pages 273 through 277.

As used herein the term "blend" means a mixture of two or more polymers while the term "alloy" means a sub-class of blends wherein the components are immiscible but have been compatibilized. "Miscibility" and "immiscibility" are defined as blends having negative and positive values, respectively, for the free energy of mixing. Further, "compatibilization" is defined as the process of modifying the interfacial properties of an immiscible polymer blend in order to make an alloy.

"Bonded carded web" refers to webs that are made from staple fibers which are sent through a combing or carding unit, which separates or breaks apart and aligns the staple fibers in the machine direction to form a generally machine direction-oriented fibrous nonwoven web. This material may be bonded together by methods that include point bonding, through air bonding, ultrasonic bonding, adhesive bonding, etc.

"Airlaying" is a well-known airforming process by which a fibrous nonwoven layer can be formed. In the airlaying process, bundles of small fibers having typical lengths ranging from about 3 to about 52 millimeters (mm) are separated and entrained in an air supply and then deposited onto a forming screen, usually with the assistance of a vacuum supply. The randomly deposited fibers then are bonded to one another using, for example, hot air or a spray adhesive. The production of airlaid nonwoven composites is well defined in the literature and documented in the art. Examples include the DanWeb process as described in US patent 4,640,810 Laursen et al. and assigned to Scan Web of North America Inc, the Kroyer process as described in US patent 4,494,278 Kroyer et al. and US patent 5,527,171 Soerensen assigned to Niro Separation a/s, the method of US patent 4,375,448 Appel et al assigned to Kimberly-Clark Corporation, or other similar methods.

"Personal care product" means products for the absorption of body exudates, such as diapers, training pants, disposable swim wear, absorbent underpants, adult incontinence products, bandages, veterinary and mortuary products, and feminine hygiene products like sanitary napkins and pantliners.

TEST METHODS

Dielectric constant and dielectric loss: The following equations are based on measurements of the dielectric constant (ϵ') and dielectric loss (ϵ'') from a solid or liquid sample using a technique requiring a Network analyzer combined with a Coaxial probe [1-7]. Unless otherwise explicitly stated the equations herein apply to equivalent volumes of material unless specific volume terms are incorporated. Generally, temperature changes relative to heating rates are derived in terms of degrees Celsius per second.

Films and solid blocks may be measured as received, while powdered and pelleted samples should be converted to films or solid blocks for measurements, or may

be evaluated using a Cavity Perturbation Method. Dielectric property values of powdered samples measured using a coaxial probe method must be corrected using a volume fraction conversion.

For comparative measurements, all powders and pellets are converted to films and solids to avoid the requirement of the volume fraction conversion. The reason for this is that powdered and rough samples create micro-air-gaps between the material under test (MUT) and the probe or electrode surface. This inherent air gap causes a decrease in capacitance, resulting in a decrease in the measured capacitance and a corresponding decrease in the resulting dielectric loss values. Rough surfaces can cause a decrease in values of from 5-20 percent, while powdered samples can result in decreases of up to 100 percent.

General computational formulas have been derived from the literature for computations relating dielectric constant and dielectric loss measurements to a set of heating rate equations. These relationships can also allow general understanding of the interrelational effects of the *Applied Electric Field* (E), and *Power* (P) of a microwave source; density and specific heats of the MUT; and the estimated relative heating rates of a target material. The algebraic relationships are described here in general form and for specific cases. All variables and terms in their respective units are defined in a single section following the equations.

20 Computational Formulas

The penetration depth (in centimeters) of an applied field of microwave energy is defined by the depth of the MUT where ~63.2 percent of the energy has been dissipated. This relationship in depth units equivalent to units used for λ_0 is given in general form as

$$D_p = \frac{\lambda_0 \{2\}^{\frac{1}{2}}}{2\pi} \left[\epsilon' \left\{ 1 + \left(\frac{\epsilon''}{\epsilon'} \right)^2 \right\}^{\frac{1}{2}} - 1 \right]^{-\frac{1}{2}} = \frac{\lambda_0 \{\epsilon'\}^{\frac{1}{2}}}{2\pi\epsilon''} \quad (1)$$

And the Half-Power Penetration Depth (in λ_0 equivalent units) representing the depth of the MUT where ~50.0 percent of the energy has been dissipated is calculated using the relationship in Equation (2).

$$D_{50} = \frac{0.347\lambda_0 \{\epsilon'\}^{\frac{1}{2}}}{\pi\epsilon''} \quad (2)$$

- 5 Note 1: A dimension similar to the D_{50} is the Radio Absorption Length which is defined as

$$l_R = \frac{\lambda_0}{2\pi\{\epsilon'\}^{\frac{1}{2}} \tan \delta}$$

Note 2: (a) A Relative Heating Rate has been determined for organic liquids under specific measurement conditions. The relative heating rate as a dimensionless number is

given as $\frac{1}{D_{50}}$. (b) Additionally, an estimated heating rate for organic liquids in $^{\circ}\text{C}\cdot\text{sec}^{-1}$

- 10 under specific conditions (200 mL water, 25 mL MUT, and 630w) is estimated by

$$\frac{3.083}{D_{50}} - 0.00254 [2].$$

The Relative Absorbed Power per unit volume is given as

$$P_v = k_p v E^2 \epsilon'' \quad (3)$$

In general, the Heating Rate Equation in $^{\circ}\text{C}$ per second for an MUT is described

- 15 by

$$\Delta T = \frac{kvE^2\epsilon''}{dC_p} \quad (4)$$

A series of calculations is made, using the measured dielectric properties of a material, to estimate the relative heating rate (in $^{\circ}\text{C}$ per second) of a material. A simplified computational formula used is

$$\Delta T = 2.389 \times 10^{-1} \cdot \frac{P_o t}{WC_p} \quad (5)$$

Note 3: A power loss (as heat) is affected by the principles of convection, radiation, and conduction as energy losses, or by direct absorption by liquids or high loss materials (e.g., water). The absorbed power (in watts) is given by the general relationship

- 5 $P_A = P_o - P_L$ where P_A is the power absorbed (in watts), P_o is the available power as initial power (in watts), and P_L is the power lost as heat (as ΔT).

Note 4: The target sample load size follows a direct relationship with the heating time and the heating rate, that is, twice the material requires twice the energy input to maintain an equivalent heating rate.

- 10 The basic microwave heating equation is as

$$P_o = 2\pi\nu\epsilon_o\epsilon' \tan \delta E^2 = 2\pi\nu\epsilon_o\epsilon'' E_i^2 \quad (6)$$

The computational form of Equation (6), with ν in GHz and E in volts·cm⁻¹, is presented as

$$P_{oi} = 5.56 \times 10^{-4} \cdot \nu \cdot \epsilon'' \cdot E^2 \quad (7)$$

So applying Equation (7) for free water at 20 °C ($\epsilon''=12.48$) and for an applied microwave

- 15 field at $\nu=2.45$ GHz we have

$$P_{oi} \approx 1.70 \times 10^{-2} \cdot E^2 \quad (8)$$

And combining Equation (5) with Equation (8) we obtain

$$\Delta T = 4.0 \times 10^{-3} \cdot t \cdot E^2 \quad (9)$$

Note 5: Solving Equation (9) for one °C per second yields an E of 15.81 volts·

- 20 cm⁻¹.

The general case for microwave heating rate is expressed as

$$\Delta T = \frac{2\pi\epsilon_{oi} t \nu \epsilon'' E^2}{k_h} \quad (10)$$

Noting that the expression

$$2\pi\epsilon_{oi}tv \quad (11)$$

has a value of 1.3622×10^{-3} for 2450 MHz, and 5.0874×10^{-4} for 915 MHz, it follows from

5 Equations (5) and (10) that the rate of change in temperature is given by

$$\frac{\Delta T}{t} = \frac{2\pi\epsilon_{oi}v\epsilon''E^2}{k_h WC_p} \quad (12)$$

Solving Equation (12) for E we obtain

$$E = \left\{ \frac{\Delta Tk_h WC_p}{2\pi\epsilon_{oi}tv\epsilon'' \tan \delta} \right\}^{\frac{1}{2}} \quad (13)$$

An additional computational form for calculating an increase in temperature

10 relative to a material, given a specific microwave field strength (E) with v in units of Hz, is given by

$$\frac{\Delta T}{t} = \frac{(8 \times 10^{-12})v\epsilon''E^2}{dC_p} \quad (14)$$

The estimated Electric Field Strength (E) given an amount of Power available for conversion to heat is given as

$$15 \quad E = \left\{ \frac{P_o k_e}{2\pi\epsilon_{oi}v\epsilon''} \right\}^{\frac{1}{2}} \quad (15)$$

Note 6: By the relationships described in Fresnel reflection we know $R_{II} + T_{II} = 1$ and $R_I + T_I = 1$.

The Loss Tangent can be used to calculate the relative Electrical Conductivity of a material under test (χ_e) and its Resistivity (ρ_e) using

$$20 \quad \frac{\epsilon''}{\epsilon'} = \frac{2\chi_e}{\epsilon'v} \quad (16)$$

And solving for (χ_e) from Equation (16) gives

$$\chi_e = \frac{\epsilon'' v}{2} \quad (17)$$

Note 7: Using Equation (17) the relative Resistivity (ρ_e) is calculated as $\rho_e = \frac{1}{\chi_e}$

Symbols and Abbreviations

c is the speed of light (as 2.9979×10^{10} cm-sec.⁻¹)

5 C_p is the specific heat of the target material (in cal.g⁻¹)

d is the density of the target material (in g-cm⁻³)

D is the power dissipation factor

D_{50} is the Half-Power Penetration Depth (in cm) representing the depth of material where 50 percent of the initial power is dissipated

10 D_p is the 63 percent-Power Penetration Depth (in cm) representing the depth of material where 63.2 percent of the initial power is dissipated

χ_e is the relative Electrical Conductivity of an MUT

E is the rms value of the applied electric field (in volts·cm⁻¹)

E_1 is the rms value of the applied electric field (in volts·m⁻¹)

15 ϵ' is designated here as the measured dielectric constant of a material (a.k.a. κ)

ϵ'' is designated here as the measured dielectric loss of a material

ϵ_0 is the dielectric permittivity of free space (as 8.86×10^{-12} farad·m⁻¹)

ϵ_{01} is the adjusted dielectric permittivity constant (as 8.86×10^{-5} farad·cm⁻¹)

ϵ_r is the complex relative permittivity

20 j is a proportionality constant

k is a unit conversion proportionality constant equal to 1.333×10^{-7}

k_e is an efficiency term constant as a fraction of 1, where 1 represents 100 percent efficiency

k_h is a proportionality constant for unit conversion equal to 4.186 for GHz and

4186 for MHz

k_P is a proportionality constant for unit conversion equal to 5.561×10^{-7}

l_R is the *Radio Absorption Length* (depth of penetration in units of the λ_0 term)

λ_0 is the wavelength (in cm) of the incident microwave energy converted using λ_0

$$5 \quad = c \text{ (in cm}\cdot\text{sec}^{-1}\text{)} \times \nu^{-1} \text{ (in Hz)}. \text{ Note: } 2450 \text{ MHz} = 12.24 \text{ cm, and } 915 \text{ MHz} \\ = 32.76 \text{ cm}$$

MUT is a Material Under Test

ν (nu) is the frequency of the applied microwave field (in MHz)

P_0 is the applied initial Power (in watts \cdot m⁻³); Note: 1 watt = 14.33 g \cdot calories \cdot min⁻¹

10 P_{0i} is a computational form of the applied initial Power (in watts \cdot cm⁻³)

P_A is the Power absorbed (in watts per unit volume)

P_L is the Power lost as thermal loss (in watts per unit volume)

P_V is the Relative Absorbed Power (as applied for a specified volume, in watts per unit volume)

15 Q is the power quality factor

R_{\perp} represents the reflected power or energy for incident energy perpendicular in orientation relative to the electronic surface vector of the MUT

R_{\parallel} represents the reflected power or energy for incident energy parallel in orientation relative to the electronic surface vector of the MUT

20 ρ_e is the relative Resistivity of the MUT

t is the time duration of an applied field (in seconds)

T_{\perp} represents the transmitted power or energy for incident energy perpendicular in orientation relative to the electronic surface vector of the MUT

T_{\parallel} represents the transmitted power or energy for incident energy parallel in

25 orientation relative to the electronic surface vector of the MUT

$\tan\delta$ is the ratio of ϵ''/ϵ' , and so $\epsilon' \cdot \tan\delta = \epsilon''$

ΔT is a unit temperature change in the target (in $^{\circ}\text{C} \cdot \text{sec}^{-1}$)

W is the weight of the sample target (in grams)

Sample calculation

- 5 It is desired to measure the broad-band dielectric relaxation spectrum for a series of specially created polymers and polymer mixtures and report dielectric properties for the commercially available microwave frequencies of 915 MHz and 2450 MHz. All samples were evaluated at 25 $^{\circ}\text{C}$. All samples as pellets or fibers were converted to polymer films of approximately 1 mm thickness before measurements. Dielectric data were archived for
- 10 the full range of 300 kHz to 3.0 GHz. Sample Numbers and designations for the special mixtures studied were as shown in Table 1.

- The measurements are made using a Network Analyzer with a low power external electric field (i.e., 0 dBm to +5 dBm) typically over a frequency range of 300 kHz to 3 GHz, although Network Analyzers to 20 GHz are readily available, for example, the HP 8720D
- 15 Dielectric Probe available from the Hewlett-Packard Company (HP). Samples are measured by placing them in contact with a coaxial reflectance probe yielding low-loss measurements for liquids, semi-solids, and solid films. Solid samples must have a substantially flat surface to ensure solid contact between the flat probe surface and the test material. The coaxial reflectance method is not recommended for powdered or
- 20 crystalline solids due to irreproducible and poor contact between the probe surface and the test material. Solid materials with high dielectric loss are also not measured well using the reflectance probe method due to precision error and sensitivity of the measurement to slight variations in the integrity of probe contact/pressure with the test material.

- The instrument is calibrated for each set of measurements using ambient air, a
- 25 short (circuit), and deionized Water (25 $^{\circ}\text{C}$). Water is then re-measured to check the calibration and the resultant dielectric constant must measure between 79 and 80 across

the range 600 kHz to 2.9 GHz. Specifically, an HP 8752C (300 kHz to 3 GHz) and HP 8720D (50 MHz to 20 GHz) Radio Frequency (RF) Network Analyzers, and an HP 85070B Reflectance Dielectric Probe have been used for dielectric determinations. Once calibrated, these instruments are used to directly measure dielectric constant, and dielectric loss factor. From this information, calculations can be made for a number of material properties, including electric modulus, power dissipation factor (loss tangent), half power penetration depth, and reciprocal half power penetration depth. These parameters, and others mentioned herein, can be used to study the dielectric properties of materials.

The Network Analyzer/reflectance probe measurement determines the real (ϵ') and imaginary (ϵ'') parts of the complex relative permittivity (ϵ_r) of a sample. The Network Analyzer measures the reflection coefficient of the MUT (Material Under Test), and an internal model in the microprocessor converts the reflection coefficient to the permittivity. The dielectric error sources include probe model accuracy (3% to 5%) and uncertainty due to the accuracy of the calibration method. The complex relative permittivity describes the interaction of a material with an applied electric field. The dielectric constant (κ) is equivalent to the complex relative permittivity (ϵ_r) by the relationship

$$\epsilon_r = \kappa = \left(\frac{\epsilon'}{\epsilon_0} \right) - j \left(\frac{\epsilon''}{\epsilon_0} \right) \quad (18)$$

where ϵ_r is the complex relative permittivity,

ϵ_0 is the permittivity in free space (i.e., 8.854×10^{-12} Farad/m)

The real part of the complex permittivity (ϵ') as measured is proportional to the dielectric constant (κ) and is a measure of the energy stored in a material when an electric field is applied across that material. This value is greater than 1 for most solids and liquids. The imaginary part of the complex permittivity (ϵ'') is called the loss factor. It is a measure of how much energy is lost from a material when an electric field is applied

across that material. This value is always greater than 0 as all materials have some loss under normal conditions. The loss factor includes the effects of conductivity of the material as well as its dielectric loss.

Computational Methods for Dielectric Measurements

5 As noted, the measured value of ϵ' is most often referred to as the *dielectric constant*, while the measurement of ϵ'' is denoted as the *dielectric loss factor*. These are measured directly using the Network Analyzer as the *Real[Permittivity]* and *Imaginary[Permittivity]*, respectively. By definition, ϵ'' is always positive, and a value of less than zero is occasionally observed when ϵ'' is near zero due to the measurement
10 error of the analyzer.

The *loss tangent* or *power dissipation factor* is defined as the calculated ratio of ϵ''/ϵ' . This loss tangent results as the vector sum of the orthogonal real (ϵ') and imaginary (ϵ'') parts of the complex relative permittivity (ϵ_r) of a sample. The vector sum of the real and imaginary vectors creates an angle (δ) where $\tan \delta$ is the analytical
15 geometry equivalent to the ratio of ϵ''/ϵ' . Thus the following relationship holds

$$\tan \delta = \frac{\epsilon''}{\epsilon'} = D = \frac{1}{Q} \quad (19)$$

where D is the power dissipation factor, and

Q is the power quality factor (often referred to in the literature)

Multiple dielectric mechanisms can contribute to the complex relative permittivity
20 (ϵ_r) of a sample. These include: dielectric constant, dielectric loss, conductivity, and various polarization effects. The sample thickness, temperature, density, homogeneity, and the like will also affect the measured dielectric properties of a material under test (MUT). An abrupt increase in dielectric loss for an MUT at a region over the measured frequency range indicates the occurrence of a dielectric transition. For example, in the
25 microwave region near 1 GHz the dipolar or rotational mechanism of molecules resonates

with the applied field; in the infrared region near 10^3 GHz the molecular vibrational mechanisms resonate with the applied field; and in the visible and ultraviolet regions near 10^6 GHz the electronic transitions resonate with an applied field. The imaginary part of the complex permittivity (ϵ'') or the loss factor exhibits an increase in value at each
5 resonance transition. The real part of the complex permittivity (ϵ') is orthogonal to the imaginary part and thus exhibits a decrease in value at the resonance points. These transitions can be measured and recorded for each MUT. For measurements in the microwave region, an abrupt increase in the loss factor indicates a resonance frequency where rotational energy is maximized relative to the applied field. Thus the application of
10 an energy field at this frequency would contribute to the maximum rotational energy and resultant conformational changes and heating.

The application of the mathematical relationships and measurements discussed allows an estimation of the microwave affinity or microwave-receptivity for a particular material subjected to a microwave field. An estimation of the absorbed power and heating
15 rate for any MUT provides information relative to selection of optimum microwave field strengths for broad applications. Selecting materials based on heating rates and required microwave power provides guidance for the composition and macro-structures of materials designed with specific microwave absorption properties.

20

DETAILED DESCRIPTION

Related material is disclosed in U.S. Patent Application Serial No. 10/034,079 entitled TARGETED BONDING FIBERS FOR STABILIZED ABSORBENT STRUCTURES by F. Abuto et al., (attorney docket No. 15,708A); in U.S. Patent Application Serial No.
25 10/034,021 entitled ABSORBENT STRUCTURES HAVING LOW MELTING FIBERS by J. Workman et al. (attorney docket No. 15,708B); and in U.S. Patent Application Serial No.

10/033,860 entitled TARGETED ON-LINE STABILIZED ABSORBENT STRUCTURES by
F. Abuto et al., (attorney docket No. 17,527) and U.S. Patent Application Serial No.
10/037,385 entitled METHOD AND APPARATUS FOR MAKING ON-LINE STABILIZED
ABSORBENT MATERIALS by T. Rymer et al., all of which were filed contemporaneously
5 herewith on December 20th, 2001 (attorney docket No. 16820). The entire disclosures of
these documents are incorporated herein by reference in a manner that is consistent
herewith.

The bonding together of nonwoven webs has been performed by a number of
processes that involve the addition of energy to the entire web or to bonding of only
10 particular points on the web. One common process is thermal bonding wherein the web is
heated until the melting point of one of the component fibers is reached. The melted fiber
bonds to other fibers in the web as it cools to give the web integrity. This process is slow
and relatively inefficient, as it involves heating the entire mass of the web to produce, in
most cases, point bonds. Thermal point bonding through the use of a patterned and anvil
15 roller system is an improvement as it delivers energy to specific points, but it remains rather
slow. These devices also require significant maintenance to keep their tolerances within
specified ranges.

The inventors have found that an energy receptive additive can be included in
synthetic fibers during production, and later excited to melt the matrix polymer and so bond
20 the web. This allows the matrix polymer to reach its melting temperature much more rapidly
than it would without the additive and allows the fiber bonding in the web to occur at faster
rate than without the additive.

In order to be industrially applicable, the energy receptive additive must absorb
energy and melt the matrix polymer at the desired frequency of electromagnetic energy
25 (between 0.01 to 300 GHz) very rapidly, in the range of fractions of a second, desirably less
than a quarter of a second and at most about half a second. Processes which involve the

absorption of energy and bonding of the web in a time as long as 30 seconds are intended to be within the scope of this invention. This melting will depend on a number of factors such as microwave generator power, additive receptivity, fiber denier, which is generally between 1 and 20, as well as the composition of the matrix polymer.

- 5 Synthetic fibers include those made from synthetic matrix polymers like polyolefins, polyamides, polycaprolactones, polyetheramides, polyurethanes, polyesters, Poly (meth) acrylates metal salts, polyether, poly(ethylene- vinyl acetate) random and block copolymers, polyethylene -b- polyethylene glycol block copolymers, polypropylene oxide-b-polyethylene oxide copolymers (and blends thereof) and any other suitable synthetic
- 10 fibers known to those skilled in the art.

- The energy receptive additive may be added to a fiber-making matrix polymer as it is compounded, or coated onto the fiber as a sizing after it is produced. A typical method of compounding the additive with the matrix polymer is with a twin screw extruder, which thoroughly mixes the components prior to extruding them. Upon extrusion, the polymer
- 15 blend is usually pelletized for convenient storage and transportation.

- If the fiber is a bicomponent fiber the energy receptive additive may be added to either or both of the parts of the fiber. The energy receptive additive may also be added to one or more components, preferably the continuous phase, of a biconstituent fiber and so intermittently distributed throughout the length and cross-section of the fiber. If the
- 20 additive to be used is not compatible with the matrix polymer into which it is to be blended, a "compatibilizer" may be added to enhance the blending. Such compatibilizers are known in the art and examples may be found in US Patents 5,108,827 and 5,294,482 to Gessner.

- Energy receptive additives can be receptive to various specific spectra of energy.
- 25 Just as a black item will absorb more energy and become warmer than the same item colored white when subjected to the same amount of solar energy, energy receptive

additives will absorb energy at their specific wavelength, directed at them. Fibers with such additives can be used as binder fibers in the production of coherent webs. One method of providing energy to a receptive material is known as dielectric heating.

Dielectric heating is the term applied to the generation of heat in non-conducting materials by their losses when subject to an alternating electric field of high frequency. The frequencies necessarily range from 0.01 to 300 GHz (billion cycles/sec). Heating of non-conductors by this method is extremely rapid. This form of heating is applied by placing the non-conducting material between two electrodes, across which the high-frequency voltage is applied. This arrangement in effect constitutes an electric capacitor, with the load acting as the dielectric. Although ideally a capacitor has no losses, losses do occur in practice and sufficient heat is generated at high frequencies to make this a viable form of heating.

The frequency used in dielectric heating is a function of the power desired and the size of the work material. Practical values of voltages applied to the electrodes are 2000 to 5000 volts/in of thickness of the work material. The source of power is by electronic oscillators that are capable of generating the very high frequencies desirable.

The basic requirement for dielectric heating is the establishment of a high-frequency alternating electric field within the material or load to be heated. Once the electric field has been established, the second requirement involves dielectric loss properties of the material to be heated. The dielectric loss of a given material occurs as a result of electrical polarization effects in the material itself and may be through dipolar molecular rotation and ionic conduction. The higher the dielectric loss (more "lossy") of a material, the more receptive to the high frequency energy it is.

As previously defined, the complex permittivity measures the ability of a material to absorb and store electrical potential energy. The real permittivity or dielectric constant (ϵ') is a measure of the relative ease of penetration of a microwave field into a material. The dielectric loss factor (ϵ'') indicates the energy storage ability of a material. The loss

tangent ($\tan\delta$) is a value indicative of a material's capacity to absorb microwave energy and convert it to heat. Loss tangents of 0.1 to 1.0 are ideal for microwave coupling for heating in this application.

For optimum coupling with a microwave field a material must exhibit a medium dielectric constant (ϵ') in concert with a high dielectric loss factor (ϵ''). The resulting loss tangent ($\tan\delta = \epsilon''/\epsilon'$) is an indicator of optimized coupling for microwave energy available for heating a material. An additional characteristic of materials readily heated using an applied microwave field is a medium to high thermal conductivity (see Equations 12-14) in concert with a dielectric loss that does not increase dramatically with temperature.

Microwave energy interacts with materials as electronic conductivity, dipole rotation and reorientation, and ionic polarization associated with far-infrared vibrational modes. Ionic polarization occurs too rapidly to affect microwave absorption and heating and so is not discussed further. Electronic conductivity can be measured as given in Equation 16. When applied in practice this value is proportional to microwave absorptivity and inversely proportional to relative Resistivity of a material (Equation 17). Materials with moderate conductivity and low Resistivity are easily heated. Resistivity values of 10^{-8} to 10^1 Ohm-meter are optimum for easy heating.

Dipole rotation and reorientation can occur with atoms of high mass due to their inherently large electron cloud. This cloud can be distorted under an electronic field creating a dipole, which is receptive to microwave absorption. Molecules with a permanent dipole will reorient in a microwave field and absorb energy. Some molecules not normally possessing a dipole will temporarily distort (or reorient) under an electric field and can absorb microwaves. In the case of certain polymers, an imposed electric field will cause reorientation and the creation of microwave absorbing dipoles.

Thus a combination of conductivity and dipole interaction will bring about microwave absorption. Metallic materials will reflect microwave energy and thus although

electrically conductive will not directly heat under a microwave field. Combinations of electrically insulating materials and polar liquids have been used to create microwave receptive materials. Oxides added to highly conductive materials or high lossy materials have also demonstrated excellent capacity to couple with microwave energy. More
5 information on this subject may be found in Microwave Processing of Materials, Publication NMAB-473, ISBN 0-309-05027-8, National Academy Press, Washington, D.C. 1994, pp. 27, 28, 30, 96.

Radio Frequency or RF heating occurs at about 27 MHz and heats by providing about half the total power delivered as ionic conduction to the molecules within the
10 workpiece, with the remainder of the power delivered as dipolar molecular rotation.

Microwave heating is dielectric heating at still higher frequencies. The predominate frequencies used in microwave heating are 915 and 2450 MHz though other frequencies may be used and particular additives may be found to be receptive at only particular frequencies. Microwave heating is 10 to 100 times higher in frequency than the usual
15 dielectric heating, resulting in a lower voltage requirement if the loss factor is constant, although the loss factor is generally higher at microwave frequencies.

Microwaves can penetrate dielectric materials and be absorbed uniformly, thereby generating heat uniformly. Microwave energy is also selectively absorbed, offering a means for self-limiting the energy taken up by heterogeneous materials, making overheating less
20 likely. These combined effects allow microwave heating to be more rapid, with less heating of surrounding materials, with a low thermal lag, and therefore with good control.

A successful energy receptive additive should have a dielectric loss factor, as noted above, that is relatively high. The energy receptive additives useful in this invention typically can have a dielectric loss factor measured in the RF or microwave frequency of between
25 about 0.5 and 15, more particularly between about 1 and 15,, and still more particularly

between about 5 and 15. It should be noted that the dielectric loss factor is a dimensionless number.

In addition to having an additive with the appropriate dielectric loss factor, successful energy receptive additive containing fibers should have flow characteristics that would enable bonding with the other fibrous components in any structure formed. It is also desirable to have the energy receptive additive containing fiber retain its shape after being activated and not melt entirely. The energy receptive additive containing fiber should also exhibit good resiliency after bonding in order to maintain the capilarity of the final structure and prevent premature wet collapse.

- 10 Examples of materials that may be suitable energy receptive additives, followed by their dielectric constants are: titanium dioxide (110), hydrogen peroxide at 0 °C (84.2), water at 20 °C (80.4), methyl alcohol at -80 °C (56.6), glycerol at 25 °C (42.5), titanium oxide (40-50), glycol at 25 °C (37), sorbitol at 80 °C (33.5), ethanol at 25 °C (24.3), propanol at 80 °C (20.1), ferrous sulfate at 14 °C (14.2), ferrous oxide at 15.5 °C (14.2),
15 calcium superphosphate (14-15), zircon (12), graphite or high density carbon black (12-15), calcium oxide granules (11.8), barium sulfate at 15.5 °C (11.4), ruby (11.3), silver chloride (11.2), silicon (11-12), hydrogenated castor oil at 27 °C (10.3), magnesium oxide (9.7), alumina (9.3-11.5), anhydrous sodium carbonate (8.4), calcite (8), mica (7), dolomite (6.8-8). Other examples include, but are not limited to, various mixed valent
20 oxides such as magnetite (Fe_3O_4), nickel oxide (NiO) and such; ferrite, tin oxide, carbon, carbon black and graphite; sulfide semiconductors such as FeS_2 , CuFeS_2 ; silicon carbide; various metal powders such as aluminum, iron and the like; various hydrated salts and other salts, such as calcium chloride dihydrate; diatomaceous earth; adipic acids; aliphatic polyesters e.g. polybutylene succinate and poly(butylene succinate-co-adipate), polymers
25 and co-polymers of polylactic acid, polymers such as PEO and copolymers of PEO, including PEO grafted with polar acrylates; various hygroscopic or water absorbing

materials or more generally polymers or copolymers or non-polymers with many sites with -OH groups; other inorganic microwave absorbers including aluminum hydroxide, zinc oxide, barium titanate and other organic absorbers such as polymers containing ester, aldehyde, ketone, isocyanate, phenol, nitrile, carboxyl, vinylidene chloride, ethylene oxide, methylene oxide, epoxy, amine groups, polypyrroles, polyanilines, polyalkylthiophenes and mixtures thereof.

A series of samples were produced to determine which of many possible additives might prove to be useful for the practice of this invention. In the samples described below, carbon black 7032 Ultra is available from the Columbia Chemical Company of Marietta, Georgia at a concentration of about 100 weight percent, magnetite M-25 is available from the Pea Ridge Iron Ore Company, Inc. of Sullivan, Missouri, at a concentration of 98.8 weight percent, silicon carbide is available from the UK Abrasives Company of Northbrook, Illinois, at a concentration of 97 to 100 weight percent, calcium chloride is available from the Sigma-Aldrich Company of St. Louis, Missouri, and titanium dioxide is available from E.I. duPont of Wilmington, Delaware. The matrix polymers, linear low density polyethylene (LLDPE) 6811A and XU 68100.34L, are available from Dow Chemical under the tradename ASPUN®. The ethylene-vinyl acetate copolymer Elvax® 410 is available from Dow Chemical. Celbond® Type 255 (T-255) fiber is a sheath/core conjugate fiber available from KoSa Inc. (Charlotte, North Carolina) with a polyethylene sheath and polyester core. In sample W, the T-255 contained 5 weight percent carbon black (CB) in the sheath. The linear low density polyethylene XU 58380.01L "metallocene" is available from Dow Chemical.

"Metallocene" polymers have a narrow polydispersity number, e.g.; Mw/Mn is 4 or less and may be produced according to the metallocene process. The metallocene process generally uses a catalyst which is activated, i.e. ionized, by a co-catalyst. The metallocene process, and particularly the catalysts and catalyst support systems are the subject of a

number of patents, such as U.S. Patent 5,374,696 to Rosen et al. and U.S. Patent 5,064,802 to Stevens et al., assigned to Dow and U.S. Patent 4,542,199 to Kaminsky.

Other constrained geometry catalysts have been used to produce similar polymer resins.

All samples were received as pellets except samples F and W, which were received
 5 as fibers. All samples were made into films of approximately 1 millimeter thickness before measurement. The composition of each sample is set out in Table 1. In Table 1, component 1 and component 2 are the polymer mixture into which the energy receptive additive (ERA) were additive. Table 2 gives the dielectric loss factor and Table 3 gives the dielectric loss tangent of the film of each sample at the frequencies of 915 and 2450 at 25
 10 °C, as tested according to the test procedure given above. All percentages in Table 1 are in weight percent of the fiber.

Table 1:

Sample ID	Description	ERA	Component 1	Component 2
A	Pellet	10% Carbon Black	20% Metallocene	70% LLDPE 6811A
B	Pellet	10% Magnetite	90% LLDPE 6811 A	----
C	Pellet	10% Silicon Carbide	90% LLDPE 6811 A	----
D	Pellet	10% Calcium Chloride	90% LLDPE 6811 A	----
E	Pellet	10% Carbon Black	90% LLDPE 6811 A	----
F	Fiber	KoSa Type 255	----	----
G	Pellet	10% Magnetite	20% EVA 410	70% LLDPE 6811A
H	Pellet	10% Carbon Black	20% EVA 410	70% LLDPE 6811A
J	Pellet	22% Carbon Black	5% Titanium Dioxide	73% LLDPE .34L
K	Pellet	11% Carbon Black	5% Titanium Dioxide	84% LLDPE .34L
M	Pellet	5% Carbon Black	95% LLDPE .34L	----
Q	Pellet	10% Silicon Carbide	20% EVA 410	70% LLDPE 6811A
R	Pellet	10% Silicon Carbide	20% Metallocene	70% LLDPE 6811A
S	Pellet	10% Calcium Chloride	20% EVA 410	70% LLDPE 6811A
T	Pellet	50% Metallocene	50% LLDPE 6811A	----
U	Pellet	10% Calcium Chloride	20% Metallocene	70% LLDPE 6811A
V	Pellet	10% Magnetite	20% Metallocene	70% LLDPE 6811A
W	Fiber	T255 w/ 5% CB Sheath	----	----

Table 2

Dielectric loss (ϵ'') for 915 and 2450 MHz at 25 °C.

Sample	915 MHz	2450 MHz
A	1.1500	0.8100
B	0.4200	0.2200
C	0.1500	0.1300
D	0.1800	0.1400
E	0.8300	0.6500
F	0.3000	0.1400
G	0.2100	1.1000
H	6.0000	3.5600
J	12.0000	8.6000
K	0.2000	0.1600
M	0.2200	0.1800
Q	0.2900	0.1900
R	0.1100	0.1000
S	0.1400	0.1400
T	0.2000	0.1500
U	0.0800	0.0600
V	0.0700	0.0900
W	0.1400	0.1400

Table 3

5 Dielectric loss tangent ($\tan \delta = \epsilon''/\epsilon'$) for 915 and 2450 MHz at 25 °C.

Sample	915 MHz	2450 MHz
A	0.25	0.1933
B	0.13	0.0719
C	0.08	0.0743
D	0.09	0.0714
E	0.17	0.1464
F	0.1	0.0461
G	0.1	0.0495
H	0.59	0.4379
J	0.6	0.7049
K	0.11	0.0899
M	0.09	0.0769
Q	0.09	0.0596
R	0.07	0.0676
S	0.07	0.0704
T	0.08	0.0655
U	0.06	0.0429
V	0.03	0.0402
W	0.05	0.0545

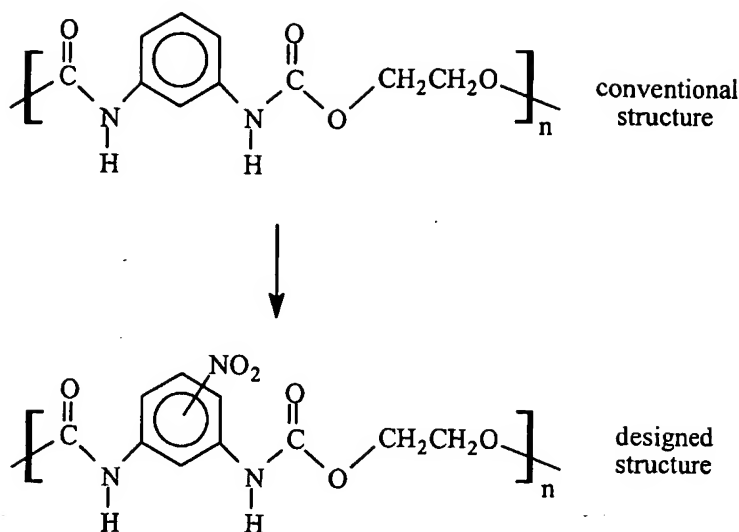
Materials J, H, A, and E, respectively exhibit enhanced microwave-receptivity where the heating rate for sample J is nearly twenty times the rate of the majority of the

materials tested. Correspondingly, these four materials require less power input to generate an equivalent heating rate.

By selecting the appropriate power settings, duration of the applied field, material composition and structure, a model of microwave heating can be deduced. Using this technique provides low cost optimization, resulting in substantial cost savings in the process of selecting energy receptive candidates. Cost reduction is achieved by reducing the viable candidate materials, structures, and microwave field settings prior to actual trial experimentation. Material candidates can be selected for optimized coupling and heating with a microwave field by using the methods taught above. Additionally, a method whereby an appropriate frequency is selected, in combination with a single material or mixtures of components, to produce a more ideal coupling and heating property may be employed. With this information, the appropriate combination of frequency and material composition may be selected yielding ideal heating properties for specialized applications.

In addition to the energy receptive additives discussed above, a number of other polymers and sensitizers may be used. Specifically selecting moieties along the polymer chain and the positioning of moieties along the polymer chain can affect the dielectric loss factor of the polymer and enhance the responsiveness of the polymer to electromagnetic energy. These include polymer composites from blend, block, graft, random copolymers, ionic polymers and copolymers and metal salts. Desirably, the presence of one or more moieties along the polymer chain causes one or more of the following: (1) an increase in the dipole moments of the polymer; and (2) an increase in the unbalanced charges of the polymer molecular structure. Suitable moieties include, but not limited to, aldehyde, ester, carboxylic acid, sulfonamide and thiocyanate groups.

The selected moieties may be covalently bonded or ionically attached to the polymer chain. As discussed above, moieties containing functional groups having high dipole moments are desired along the polymer chain. Suitable moieties include, but are



not limited to, urea, sulfone, amide, nitro, nitrile, isocyanate, alcohol, glycol and ketone groups. Other suitable moieties include moieties containing ionic groups including, but are not limited to, sodium, zinc, and potassium ions.

For example, a nitro group may be attached to an aryl group within the polymer chain. It should be noted that the nitro group may be attached at the meta or para position of the aryl group. Further, it should be noted that other groups may be attached at the meta or para position of the aryl group in place of the nitro group. Suitable groups include, but are not limited to, nitrile groups. In addition to these modifications, one could incorporate other monomer units into the polymer to further enhance the responsiveness of the resulting polymer. For example, monomer units containing urea and/or amide groups may be incorporated into the polymer.

Suitable moieties include, but are not limited to, aldehyde, ester, carboxylic acid, sulfonamide, alcohols, glycols and thiocyanate groups. However, other groups having or enhancing unbalanced charges in a molecular structure can also be useful; or a moiety having an ionic or conductive group such as, e.g., sodium, zinc, and potassium ions. However, other ionic or conductive groups can also be used.

Specific combinations include low density PE/polyethylene-polyvinylacetate block copolymer, LDPE/polyethylene glycol, PE/ polyacrylates, polyethylene-vinyl acetate copolymer, polyester, polyurethane, polyacrylates, polyethylene glycol (PEG), polyacrylamide (PAA), polyethylenimine (PEEM), polyvinyl acetate (PVAC), polyvinyl alcohol (PVA), polymethylacrylic acid- sodium salt (PMA-Na), polyacrylic acid sodium salt (PA-Na), and poly (styrene sulfonate-co-methyl acrylic acid) sodium salt (P (SS-co-MA)-Na), NaCl, CaCl₂, MgCl₂, Na₂CO₃, Mg acetate, Al acetate and Al (iso-peroxide) and polymer of terephthalic acid, adipic acid and 1, 4 butanediol, and polybutylene succinate copolymers..

10 As will be appreciated by those skilled in the art, changes and variations to the invention are considered to be within the ability of those skilled in the art. Examples of such changes and variations are contained in the patents identified above, each of which is incorporated herein by reference in its entirety to the extent consistent with this specification. Such changes and variations are intended by the inventors to be within the

15 scope of the invention.

What is claimed is:

1. A fiber comprising an energy receptive additive capable of dielectric heating and wherein said fiber has a dielectric loss of between 0.5 to 15.
2. The fiber of claim 1 and wherein said fiber has a dielectric loss of between 1 to 15.
- 5 3. The fiber of claim 1 and wherein said fiber has a dielectric loss of between 5 to 15.
4. The fiber of claim 1 having a dielectric loss tangent of between 0.1 to 1.
5. The fiber of claim 4 having a dielectric loss tangent of between 0.3 to 0.7.
6. The fiber of claim 3 further comprising a synthetic polymer selected from the group consisting of polyolefins, polycaprolactones, polyamides, polyetheramides,
10 polyurethanes, polyesters, poly (meth) acrylates metal salts, polyether, poly(ethylene-vinyl acetate) random and block copolymers, polyethylene -b- polyethylene glycol block copolymers, polypropylene oxide-b-polyethylene oxide copolymers and blends thereof.
7. The fiber of claim 3 wherein said energy receptive additive is selected from the group
15 consisting of carbon black, ferrite, tin oxide, silicon carbide, calcium chloride, zircon, magnetite, silicon carbide, calcium chloride, alumina, magnesium oxide, and titanium dioxide.
8. The fiber of claim 5 wherein said energy receptive additive is present in an amount between 2 and 40 weight percent.
- 20 9. The fiber of claim 6 wherein said energy receptive additive is present in an amount between 5 and 15 weight percent.
10. The fiber of claim 7 wherein said fiber is a bicomponent fiber selected from the type consisting of sheath/core and island in the sea.
11. The fiber of claim 8 wherein said fiber is a sheath/core bicomponent fiber and said
25 additive is present in said sheath.

12. The fiber of claim 8 wherein said fiber is a sheath/core bicomponent fiber and said additive is present in said core.
13. The fiber of claim 8 wherein said fiber is a sheath/core bicomponent fiber and said additive is present in said sheath and said core.
- 5 14. The fiber of claim 7 wherein said fiber is a biconstituent fiber.
15. The fiber of claim 8 wherein said fiber is crimped.
16. The fiber of claim 8 wherein said fiber is extendible.
17. The fiber of claim 8 wherein said fiber is elastic.
18. The fiber of claim 11 wherein said energy receptive additive is carbon black in an
10 amount between 2 and 40 weight percent.
19. A fiber comprising an energy receptive additive in an amount between 5 and 15 weight percent, synthetic polymer and wherein said fiber has a dielectric loss of at least 0.5.
20. A nonwoven web comprising fibers having an energy receptive additive capable of dielectric heating and having a dielectric loss of between 0.5 to 15.

CHARTER

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